Progress Report
Investigation of Thermal Fatigue in Fiber Composite Materials

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by

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INTRODUCTION

The object of this research program is to provide information on the capabilities and limitations of graphite fiber-epoxy materials to serve under thermal fatigue conditions and to make possible the production of improved fiber composites through a better understanding of the influence of fiber orientation and lamination sequence on their thermal fatigue performance.

The initial time was spent procuring equipment, fabricating a suitable sample mold, and preparing samples. Subsequently, the composite specimens were tested in the as fabricated condition for elastic modulus, tensile strength, and thermal expansion coefficients.

Presently, two different pieces of equipment are being developed to thermally cycle the samples.

FABRICATION

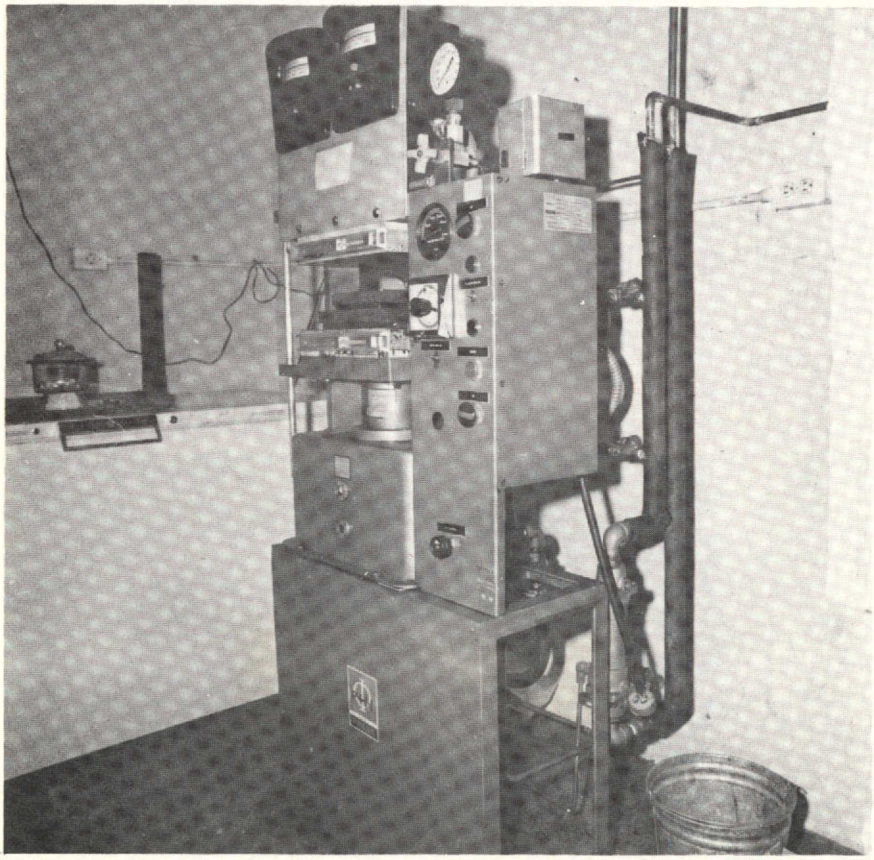
Samples were made from epoxy preimpregnated graphite sheets obtained from Monsanto. These sheets consisted of Monsanto resin type 4617/mPDA with a 55 w/o fiber. The sheets, originally 2' x 4', were cut into 6" squares. The squares were cut so that the fiber axis was at 0° or 90° with the edge of the square for the unidirectional and the -45°, +45° samples and at 0°, 30°, and 60° for the 0°, 30°, 60°, 90° sample.

Twelve layer samples were made for each of the following orientation sequences:

	Layer Number	1	2	3	4	5	6	7	8	9	10	11	12
Angle in degrees	I	0	0	0	0	0	0	0	0	0	0	0	0
	II	+45	-45	+45	-45	+45	-45	+45	-45	+45	-45	+45	-45
	III	0	30	60	90	-60	-30	-30	-60	90	60	30	0

The layers were then placed in a mold fitted with a die and punch. The mold was then placed between the platens of the hot press where a pressure of 50 psi and a temperature of 80°C were applied (Figure 1). At the end of two hours the pressure was increased to 100 psi and the temperature was increased to 150°C. The samples were then cured an additional two hours. After the samples were properly cured the hot press was turned off and the samples were cooled to room temperature by water flowing through the platens.

Once the samples were removed from the mold, they were checked for any visible flaws and the thickness was measured. The final sample measured 6" x 6" x 0.080".



Hot Press

Figure 1

MICROSCOPIC EXAMINATION

To prevent time being wasted by testing poor samples, each sample was examined microscopically. Two cross sections of each samples, cut at right angles to one another, were examined under the microscope. The purpose of the examination was to ascertain the fiber orientation in each layer, to check thickness, and parallelism of the individual layers as well as the uniformity of fiber distribution and general integrity of the composite.

MODULUS OF ELASTICITY

The proposed work in this area consisted of measuring the elastic modulus in the 0° direction of the three different composite configurations. Later it was decided that a complete characterization of the material was needed. This characterization required measuring the longitudinal modulus (E_L), the transverse modulus (E_T), the shear modulus (G_{LT}), and Possion's ratio (ν_{LT}) of the unidirectional sample. From these values E_L, E_T, G_{LT}, ν_{LT} the elastic modulus of any composite with any construction can be calculated and compared to experimental results.

Specimens, approximately 0.5" x 3.0" x 0.08", were cut from the samples. To insure that an even load was applied to the specimens and that the specimens were not damaged by the jaws of the testing machine, aluminum strips were glued to each side of the sample with epoxy at both ends.

All samples were loaded in tension with the load and strain being recorded. The accumulated strains were recorded by the use of SR-4 strain gages and a strain gage recorder, both produced by BLH Electronics. The strain gages were attached to the unidirectional specimens so that the gage was at 0° with the fiber axis to measure ε_L and at 90° to measure ε_T. Two measurements were taken on the -45°, +45°, one with the strain gage oriented in either the -45°, or +45° direction and the other along the 0° axis. The applied load was read directly from the Tinus-Olsen Testing Machine.

Due to the small loads required to break the unidirectional 90° specimens, the modulus of elasticity was obtained from a four point flexure test. The strain was obtained from strain gages and the applied load from loads placed on the testing apparatus.

Possion's ratio was calculated using a dual strain gage that was attached to the unidirectional specimens. The shear modulus was measured using the method of torque applied to a rectangular bar.

DETERMINATION OF THERMAL EXPANSION

The coefficient of thermal expansion was determined for each panel with different construction. One specimen was cut from the -45°, +45° and the 0°, 30°, 60°, 90° panels since the thermal expansion should be the same in all directions in these two panels.[3] The specimens from the unidirectional samples were taken such that the following angles were made with the unidirectional axis:

Angle in Degrees 0, 15, 30, 45, 60, 75, and 90

The ends of the specimens were ground flat, parallel and perpendicular to the specimen axis.

Each specimen was slowly heated in a dialatometer, Figure 2, from room temperature to 180°C at the approximate rate of 1°C/minute. The specimens were then cooled to room temperature by cutting off the heating current. While the first heating and cooling curves showed hysteresis, further heating followed very closely the cooling curve and the behavior became essentially reversible. The thermal expansion coefficient was determined from the cooling cycle and was measured between 50°C and 150°C.

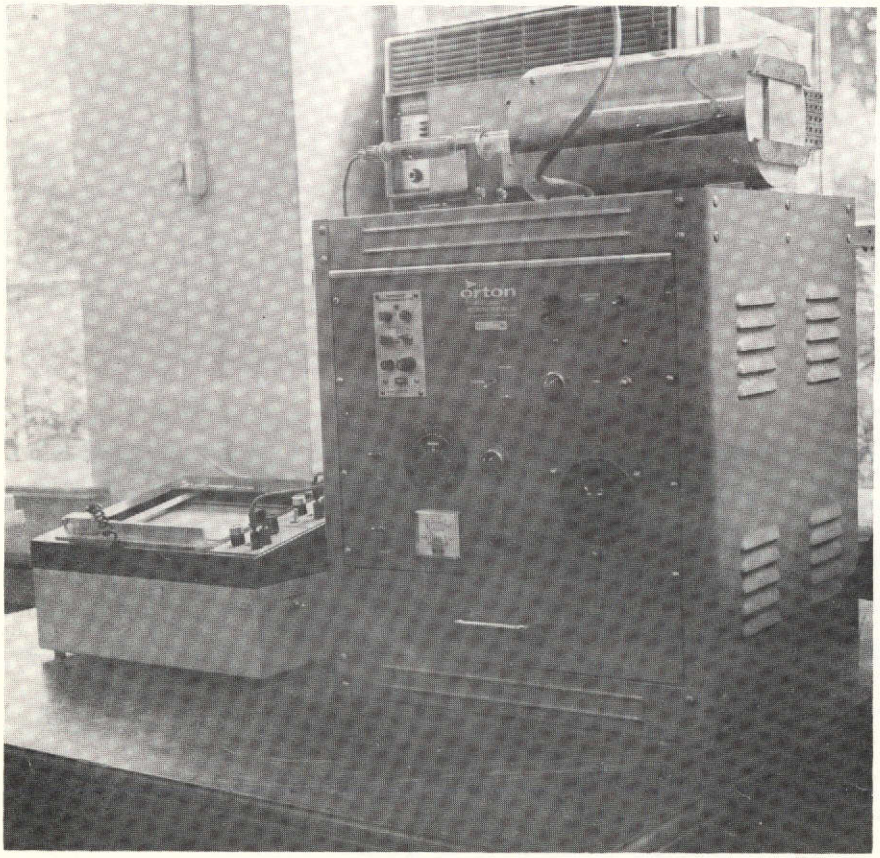


Figure 2

ULTIMATE TENSILE STRENGTH

Samples used to calculate elastic modulus were loaded until fracture to obtain the ultimate tensile strength.

RESULTS AND DISCUSSION

Microscopic Examination

Figures 3, 4, and 5 show the photomicrographs of the fiber composites.

Figure 3 is the unidirectional sample. The top micrograph was taken at 90° to the fiber axis. Four fiber layers are distinguishable even though there is good bonding between and good fiber distribution in the layers. The second two micrographs were taken at 0° to the fiber direction. The fibers are essentially parallel.

Figure 4 is a micrograph of the -45°, +45° sample. There is good bonding between layers and little or no transverse cracking.

Figure 5 is of the 0°, 30°, 60°, 90° sample. The top micrograph shows seven layers starting with the 0° direction and ending with two 30° directions. The six layers on top represent one-half of the thickness (the mid plane is marked on the photomicrograph by a dark line. The construction of the bottom half is identical to that of the upper half). The other two micrographs taken at higher magnification show the fiber cross sections in different layers. Good bonding with practically no cracking was observed for this configuration.

Modulus of Elasticity

a) Theoretical Considerations[2]

For a single ply fiber composite, the modulus of elasticity, E, when the uniaxial tensile axis is along the fiber direction is given by the equation:

$$E_L = E_f V_f + E_m(1-V_f) \tag{1}$$

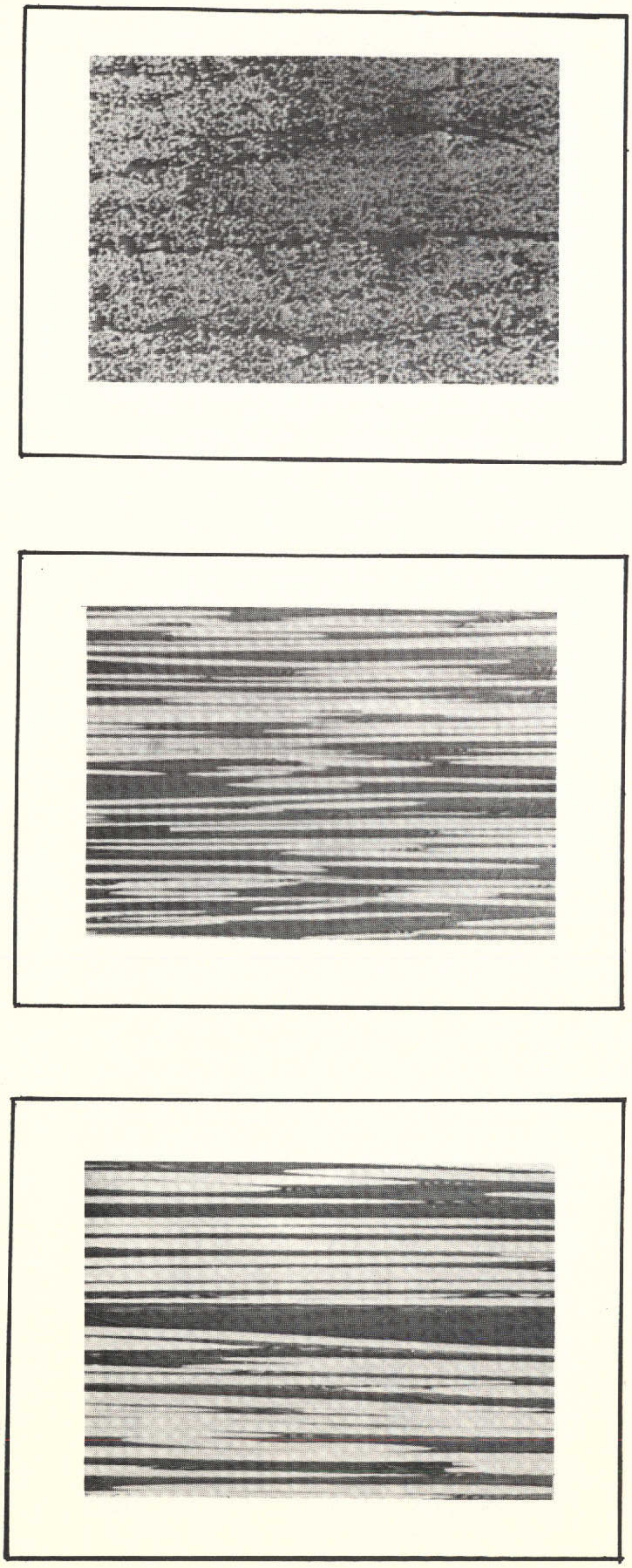


Figure 3

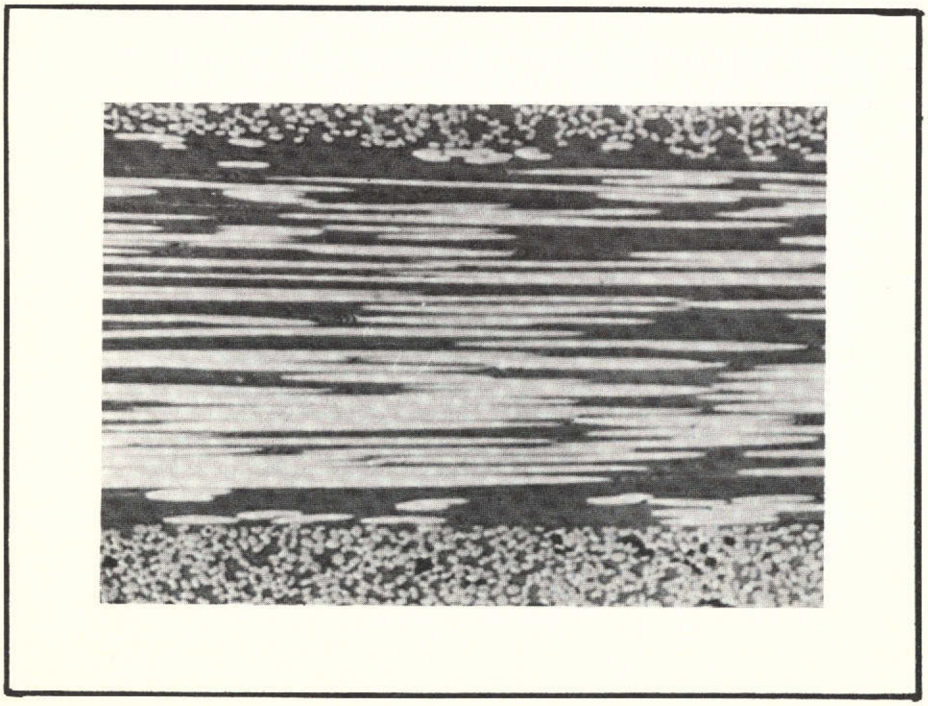


Figure 4

Figure 5

and when the uniaxial tensile axis is normal to the fiber direction, E is given by:

$$E_T = \frac{E_m E_f}{E_f(1-V_f) + E_m V_f} \tag{2}$$

Where:

E_L = longitudinal modulus

E_T = transverse modulus

V_f = volume fraction fiber

V_m = volume fraction matrix

If then a value of the shear modulus (G_{LT}) and Possion's ratio (ν_{LT}) is obtained by experimental results, the composite can be fully characterized.

The shear modulus (G_{LT}) is obtained from the torque equation for a rectangular bar:

$$G_{LT} = \frac{M_t}{K_1 \theta (2a)^3 (2b)} \tag{3}$$

Where:

M_t = (load)(distance) i.e. twisting moment

θ = (angle of twist) ÷ (length of sample)

a - 0.5 thickness of sample

K_1 = constant obtained from b/a

Possion's ratio can be calculated from the results obtained when strain, ε, is measured in both the longitudinal and transverse directions for the uniaxial composite.

$$\nu_{LT} = \frac{\varepsilon_t}{\varepsilon_L} \tag{4}$$

It also holds that:

$$\frac{\nu_{TL}}{\nu_{LT}} = \frac{E_T}{E_L} \tag{5}$$

In deriving stress-strain relationships for a single layer of a laminated composite, it is assumed that the stress normal to the layer is negligible. Thus it results in a plane stress state.

The stress-strain relationship for a lamina in the matrix form is:

$$\begin{bmatrix} \sigma_L \\ \sigma_T \\ \tau_{LT} \end{bmatrix} = \begin{bmatrix} Q_{11} & Q_{12} & 0 \\ Q_{12} & Q_{22} & 0 \\ 0 & 0 & Q_{66} \end{bmatrix} \begin{bmatrix} \varepsilon_L \\ \varepsilon_T \\ \gamma_{LT} \end{bmatrix} \tag{6}$$

Where the components of the stiffness matrix, Q, are:

$$\begin{aligned} Q_{11} &= \frac{E_L}{1-\nu_{LT}\nu_{TL}} \\ Q_{22} &= \frac{E_T}{1-\nu_{LT}\nu_{TL}} \\ Q_{12} &= \frac{\nu_{LT}E_L}{1-\nu_{LT}\nu_{TL}} \\ Q_{66} &= G_{LT} \\ Q_{16} &= Q_{26} = 0 \end{aligned} \tag{7}$$

When the stresses are in an arbitrary coordinate system (x, y, z), the following matrix is obtained:

$$\begin{bmatrix} \sigma_X \\ \sigma_Y \\ \tau_{XY} \end{bmatrix} = \begin{bmatrix} \bar{Q}_{11} & \bar{Q}_{12} & 0 \\ \bar{Q}_{12} & \bar{Q}_{22} & 0 \\ 0 & 0 & \bar{Q}_{66} \end{bmatrix} \begin{bmatrix} \varepsilon_X \\ \varepsilon_Y \\ \gamma_{XY} \end{bmatrix} \tag{8}$$

Where the components of the stiffness matrix, $\overline{Q}$, which are now referred to an arbitrary set of axes, are given by:

$$\begin{aligned}
\overline{Q}_{11} &= Q_{11}\cos^4\theta + 2(Q_{12} + Q_{66})\sin^2\theta\cos^2\theta + Q_{22}\sin^4\theta \\
\overline{Q}_{22} &= Q_{11}\sin^4\theta + 2(Q_{12} + Q_{66})\sin^2\theta\cos^2\theta + Q_{22}\cos^4\theta \\
\overline{Q}_{12} &= (Q_{11} + Q_{22} - 4Q_{66})\sin^2\theta\cos^2\theta + Q_{12}(\sin^4\theta + \cos^4\theta) \\
\overline{Q}_{66} &= (Q_{11} + Q_{22} - 2Q_{12} - 2Q_{66})\sin^2\theta\cos^2\theta + Q_{66}(\sin^4\theta + \cos^4\theta)
\end{aligned} \tag{9}$$

From these equations it is possible to calculate $\overline{Q}_{ij}$ for each layer of the composite.

The laminate constitutive equations are then used to determine the elastic modulus of a multilayer composite.

$$\begin{bmatrix} N_X \\ N_Y \\ N_{XY} \end{bmatrix} = \begin{bmatrix} A_{11} & A_{12} & A_{16} \\ A_{12} & A_{22} & A_{26} \\ A & A_{26} & A_{66} \end{bmatrix} \begin{bmatrix} \varepsilon_X \\ \varepsilon_Y \\ \gamma_{XY} \end{bmatrix} \tag{10}$$

Where:

$$A_{ij} = hp \quad (Q_{ij})_n \; {}^{n=x}_{n=1}$$

hp = thickness of one layer

X = total number of layers

N = force

The modulus of elasticity can be obtained from the equation:

$$E = \frac{1}{h}\left(A_{11} - \frac{A_{12}^2}{A_{22}}\right) \tag{11}$$

h = total thickness of composite

Using the following values, the elastic modulus of the different fiber orientation was calculated:

$E_L = 26.44 \times 10^6$ psi

$E_T = 0.99 \times 10^6$ psi

$G_{LT} = 0.46 \times 10^6$ psi

$\nu_{LT} = 0.33$

<u>For the -45°, +45° sample:</u>

Along the fiber axis (either -45°, +45°) $E = 13.75 \times 10^6$ psi

Along the 0° axis $E = 1.98 \times 10^6$ psi

<u>For the 0° - 30° - 60° - 90° sample:</u> $E = 9.03 \times 10^6$ psi

b) Experimental Data

The strain and load data taken during the testing was plotted and a best fit line was drawn. From this the modulus, E, was calculated using:

$$E = \frac{\frac{\text{load}}{\text{area}}}{\text{strain}} \tag{12}$$

This was done on all specimens except the transverse unidirectional specimen where four point bending was used. The modulus in this case is given by the formula:

$$E = \frac{3P}{2bh^2\varepsilon} \tag{13}$$

Where:

E = elastic modulus

P = applied load

b = width of specimen

h - thickness of specimen

ε = strain

and the distance between knife edges in the four point bending test was 2".

Using formulas (1) and (2) the modulus for the unidirectional sample was calculated. E_L was 27.73 X 10^6psi and E_T was 1.10 X 10^6psi. These values compared very favorably with the E_L of 26.44 X 10^6psi and E_T of 0.99 X 10^6psi obtained experimentally. The experimental values were used in the theoretical calculations.

The -45°, +45° specimen had a modulus of 11.32 X 10^6psi when measured at 0°. These values compared well with the 13.75 X 10^6psi and the 1.98 X 10^6psi moduli calculated.

The 0°, 30°, 60°, 90° specimen should have the same modulus in all directions. The modulus measured was 8.3 X 10^6psi as compared to the calculated value of 9.03 X 10^6psi.

The calculated values were higher than the experimental values in most cases. This could be due in part to the specimen not being tested exactly along the designated axis. It is easy to see the dramatic reduction in modulus in the -45°, +45° specimen when the tensile axis is moved away from the fiber axis.

THERMAL EXPANSION

The thermal expansion coefficient α of the quasi-isotropic samples (i.e. -45°, +45° and the 0°, 30°, 60°, 90°) were very close. The value of this coefficient α for the (-45°, +45°) sample was 1.6 X 10^{-6}/°C as compared to 2.2 X 10^{-6}/°C for the (0°, 30°, 60°, 90°) sample. Considering the values are subject to an experimental error as much as ± 0.5 X 10^{-6}/°C, the values could be considered to be the same. It can be seen that these values of the coefficients are very small when one considers that a unidirectioned composite of the same material and proportions yields a transverse coefficient in the range of 70 X 10^{-6}/°C.

The unidirectional sample showed a clear trend for the expansion coefficient to increase as the angle between the fiber axis and specimen axis increased from 0° to 90°. This is shown in Figure 6. The simple formula for a homogeneous orthotropic material is $\alpha_c = \alpha_{0°}\cos^2\theta + \alpha_{90°}\sin\theta$ and the experimental results show a good correlation with this formula.

Coefficient of Thermal Expansion
Unidirection Sample

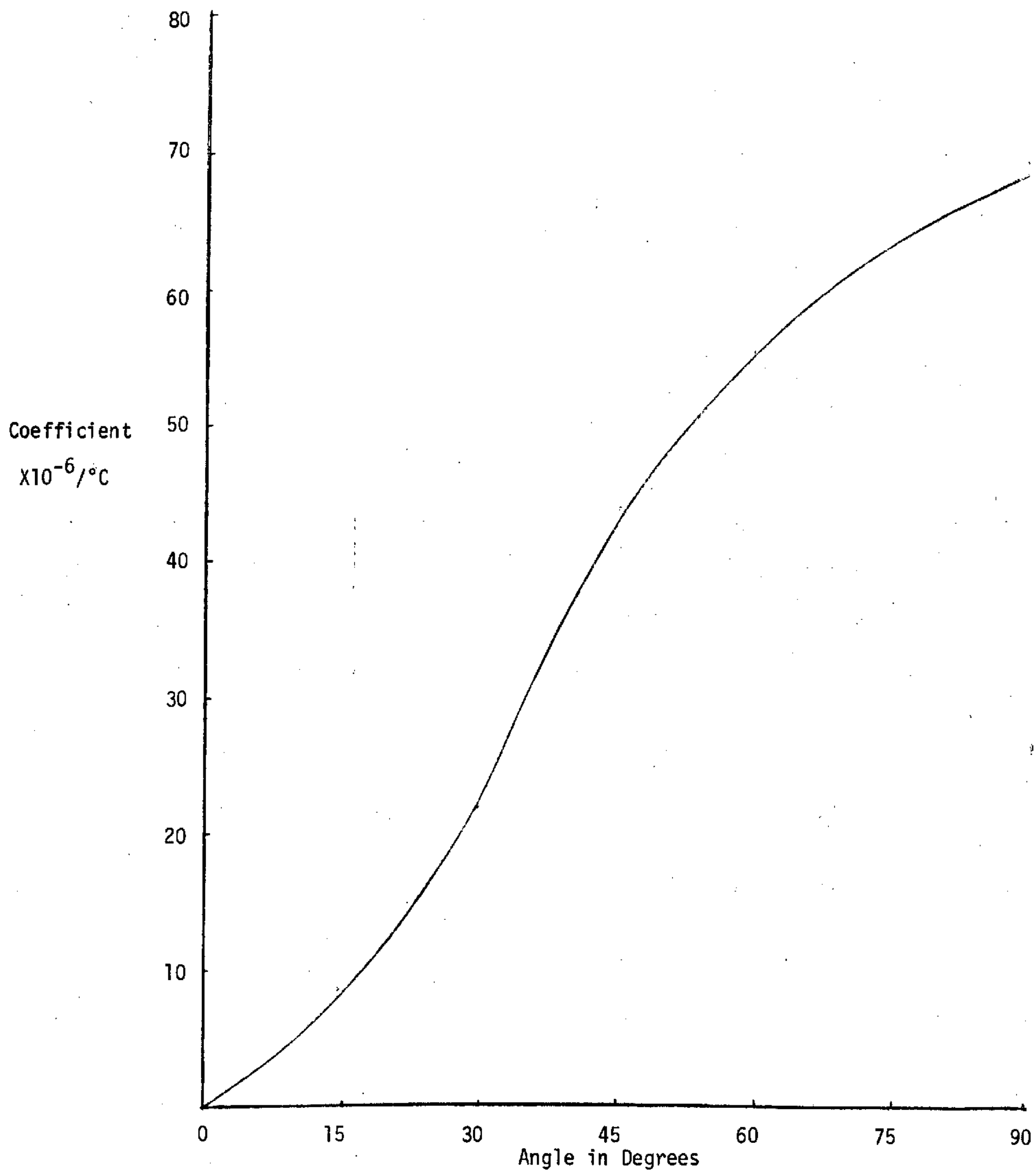


Figure 6

ULTIMATE TENSILE STRENGTH

The ultimate tensile strength of the unidirectional longitudinal specimen was 34,000 psi while the transverse specimen was 3,645 psi. The (-45°, +45°) sample had a tensile strength of 27,400 psi along the fiber axis and 15,840 psi at 45° from the fiber axis. The (0°, 30°, 60°, 90°) specimen had a uniform tensile strength of 24,300 psi.

SUMMARY AND CONCLUSIONS

1. The modulus of elasticity of the composites is as follows.

Unidirectional 0° axis	26.44×10^6 psi
Unidirectional 90° axis	0.99×10^6 psi
-45°, +45° along fiber axis	13.75×10^6 psi
0°, 30°, 60°, 90°	9.03×10^6 psi

2. Microscopic examination of the composites shows little cracking after fabrication.

3. The thermal expansion coefficient of the quasi-isotropic samples were very close. The unidirectional sample follows the expansion formula $\alpha_C = \alpha_{0°}\cos^2\theta + \alpha_{90°}\sin^2\theta$.

PLAN FOR FUTURE WORK

1. Development of Thermal Cycling Apparatus
2. Thermal Cycling of Material
3. Determination of thermal expansion coefficient, modulus, and strength of all cycled samples
4. Calculation of thermal stresses developed
5. Examination of fractured surface by the use of a scanning electron microscope
6. Evaluation of all data obtained

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